X-ray Diffraction System for Measuring Strains in Films

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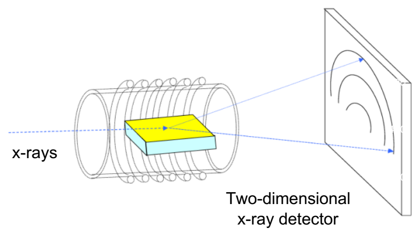
SCIENTIFIC ACHIEVEMENT

We have developed a facility utilizing x-ray synchrotron radiation to measure strains, in-situ, in thin films attached to, and constrained by, a relatively massive substrate. In this facility, strain measurements can be acquired in-situ at temperatures up to 1100 °C in air. A system modification, currently under development, will incorporate an environmental cell to permit measurements in a controlled atmosphere. The system utilizes a highly collimated x-ray beam incident on the sample at a small incidence angle. A part of the Debye ring pattern from the polycrystalline film which is diffracted into the upper half plane is recorded using a large area detector. Strain in the film (e.g., an in-plane compressive strain with associated out-of plane expansion) causes an elliptical distortion of the Debye rings. The distortion is analyzed to determine the strain in the film. Since the entire recorded pattern can be analyzed, a very high level of precision is achievable (about 10^{-5} in strain). The system is designed to accept a convenient sample size and permits rapid sample changes. Relatively few high temperature x-ray diffraction measurements of strains in constrained polycrystalline films have been reported. Typically, systems developed for this purpose require special sample sizes and/or configurations, and achievable precision in the strain measurements is significantly lower than obtained here. Generally, only a few θ-2θ spectra taken at several emission angles relative to the sample surface normal are utilized to measure elliptical distortion. (Here, entire Debye arcs are utilized). With its high precision and accuracy, and with its relative ease of use and rapid measurement capability, this new facility is capable of providing important new information pertaining to strains in polycrystalline films.

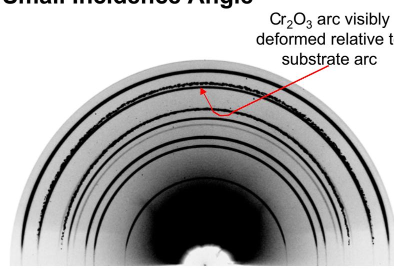
SIGNIFICANCE

This system was designed for the primary purpose of measuring growth and residual strains in oxides that form naturally on metallic alloys in high temperature oxidizing environments. The elucidation of growth strains is particularly valuable for determining growth mechanisms and their modification with alloying agents. A knowledge of these growth mechanisms is needed to develop improved protective oxides for systems operating in high temperature environments, such as turbine engines for aircraft and land based energy generation. More generally, however, the system will permit in-situ measurements of strains in controlled environments, in a variety of constrained polycrystalline films. For example, strain variations which occur in polycrystalline films deposited on dissimilar substrates and exposed to temperature excursions could be measured with precision as the sample is thermally cycled. Such strains result from thermal expansion differences between film and substrate and can seriously affect mechanical and other properties of systems undergoing temperature swings (e.g., solid oxide fuel cells).and may lead to new methods for controlling the properties of the natural protective layer.

Polycrystal Diffraction at Small Incidence Angle



Schematic of system for obtaining a Debye diffraction pattern from a constrained polycrystalline film. The sample rests in a furnace.



Diffraction pattern from Cr_2O_3 film thermally grown on Fe-Cr-Ni substrate. The elastic strain in the film is determined from the elliptical distortion of rings caused by in-plane compression and out-of-plane expansion of the oxide.

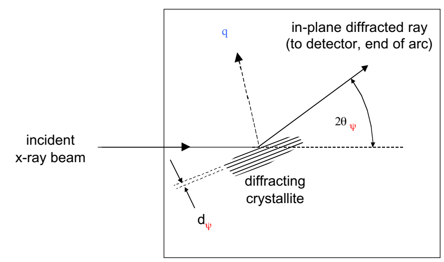
MEASURING GEOMETRY

A specimen is illuminated by x-rays at a small angle of incidence. Those crystallites in the specimen that happen to be oriented in such a way as to satisfy the Bragg diffraction condition

$$n\lambda = 2\, d_{\psi} \sin\theta_{\psi}$$

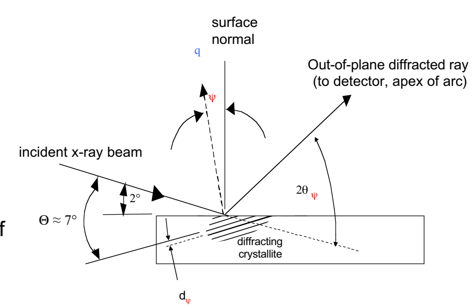
for the illuminating x-rays and for a particular crystallite d-spacing cause x-rays to be diffracted into well-defined Debye arcs. All of the diffracted radiation that escapes the specimen in several Debye arcs impinges on a two-dimensional detector. Thus data are simultaneously collected over the range of ψ angles spanned by the arcs, typically about 75 degrees; there is a corresponding range of d-spacings d_{ψ}. The deformation (deviation from circularity) of an arc is analyzed to determine the d_{ψ} and thus measure the strain of the polycrystalline film.

Top view



The surface normal is out of the plane of the drawing; ψ, the angle between the surface normal and the scattering vector q, is maximal; d_{ψ} sustains in-plane strain.

Side view



The surface normal is in the scattering plane; ψ, the angle between the surface normal and the scattering vector q, is minimal; d_{ψ} exhibits out-of-plane relaxation.

BES - DOE

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